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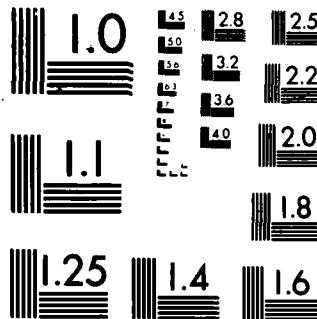
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POLYDIOXOLANE POLYMER ELECTROLYTE

By

J. S. Foos and S. M. Erker

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POLYDIOXOLANE POLYMER ELECTROLYTE

J. S. Foos and S. M. Erker

There has been much interest in an all solid-state battery incorporating a polymer electrolyte (PE). Polymer electrolytes of most current interest are composed of polyethylene or polypropylene oxide (PEO or PPO) and lithium salts (1-5). These electrolytes have conductivities of $\sim 10^{-5}$ S/cm at temperatures between 55 and 85°C.

In past work on liquid electrolytes for secondary Li batteries, we observed that samples of dioxolane containing LiAsF₆ appeared to spontaneously polymerize giving an optically clear polymeric material. The expected structure of this polymer $[-CH_2CH_2OCH_2O-]_n$ contains the -C-C-O sequence that occurs in PEO (6) and therefore might be expected to form complexes with alkali metal salts. It was later observed that LiAsF₆/dioxolane solutions could be stabilized by treatment with Li. Such samples remained liquid at 70°C but would polymerize rapidly when exposed to air. It was proposed that the polymerization was initiated by oxidation which produced the carbocation initiators typically implicated in dioxolane polymerization (6). A recent patent relates that acid also is an initiator for such polymerization and that treatment with base is also stabilizing (7).

The above observations suggest that the polymerization of the stabilized LiAsF₆/dioxolane might be initiated by electrochemical (EC) oxidation producing the required acid and carbocation initiators. Thus a PE might be formed in situ allowing improved interfacial contact with electrode materials, the formation of very thin electrolyte films on electrodes, and the addition of electrolyte to the cell late in assembly. Herein is reported preliminary studies of the formation and conductivity of the polydioxolane/LiAsF₆ polymer electrolyte.

EXPERIMENTAL

The synthesis of the polymer electrolytes and cell construction were carried out under an Ar atmosphere. The dioxolane (Aldrich, gold label) was distilled from sodium benzophenone ketyl. The LiAsF₆ (U.S. Steel Agrichemicals) was used as received. The electrolytes were made using cooled dioxolane containing Li metal as an acid and carbocation scavenger.

Some initial AC conductivities were done using an impedance bridge (Gen Rad 1650-B). Subsequently the conductivities were calculated from complex impedance plots using the real component values at imaginary component minima (8). Measurements were made over a 50 Hz to 100 kHz range with a lock-in amplifier (PAR 5204) using a method similar to one previously described (9). The cell consisted of two stainless steel (SS) electrodes held in contact with the PE. DC conductivities were measured between two Li foil electrodes supported on metal substrates. The cell was cycled at constant current and the conductivity calculated from the plateau voltages measured.

PE Synthesis.—The PE was synthesized using chemical and EC initiation:

Chemical oxidation initiation.—A trace amount of dichlorodicyano benzoquinone (DDQ) (10-20 mg) was added to 5 ml of 2.5M LiAsF₆ in dioxolane. The solution became orange due to the DDQ but no immediate reaction was observed. After 2 h at rt, the solution was polymerized. Longer times gave polymers with increased rigidity. **EC oxidation initiation.**—The general technique is to apply a current or voltage pulse between SS electrodes. At 3.0-3.2V (vs. Li), see Fig. 1, the dioxolane is oxidized at the anode and Li plated at the cathode. This initiates the growth of polymer at the anode. The cell is allowed to set for a measured period of time and, in

certain experiments, the film covered anode is then withdrawn from the bulk electrolyte. Longer setting times give thicker films. In experiments which use very thin cells, e.g., 0.025 cm, the polymerization continues across the entire cell and the conductivity measurements are made on the PE in the synthesis cell.

EC Cells Used in PE Synthesis.—Cell I is illustrative of the cells used in potentiostated initiation experiments. The cell consists of a prismatic glass container (4 cm w x 1 cm d x 6 cm h) containing SS working and counter electrodes, and a Li reference electrode. After polymerization, the working electrode (anode) is removed from the cell and the PE can be isolated from the substrate. If the cell is allowed to set for a long period of time after initiation (e.g., a day) and before electrode removal, then much of the electrolyte may become gelled even across a 1 cm cell. Generally films made in this manner were removed from the cell shortly after initiation and were ~ 0.1 cm thick.

In a second cell, Cell II, the thickness of the film produced is controlled by the thickness of the cell. This cell consists of two heavy SS electrodes separated by a U-shaped polymer spacer, typically 0.025 cm thick, that serves to define the cell thickness, to insulate one electrode from the other, and to contain the liquid electrolyte prior to polymerization. This cell gives thin PE films and additionally serves as an AC conductivity cell for PE's formed in it.

RESULTS AND DISCUSSION

Precursor Electrolyte Preparation.—Solutions of LiAsF₆ in dioxolane were prepared in several concentrations (1.0-2.5 m (molal)) with saturation at ~ 3.3 m (~ 2.9 M) at rt. The spontaneous polymerization observed in earlier work was not apparent. This may be due to the special efforts made to exclude impurities, however, the dioxolane used was of higher quality as purchased (Aldrich gold label) than that used previously (Aldrich 99%). The solutions appeared stable at rt for days (or even weeks) even after brief exposure to air.

Chemical Preparation of the PE.—DDQ was added to 2.5M LiAsF₆ in dioxolane giving polymerization in 2 h. Although the polymerization was rapid, the polymer was not examined for more than 24 h, after which time the polymerization was essentially complete. The PE was found to be rubber-like, i.e., deforming under an applied load and recovering its original shape when the load was removed. It was difficult to cut even with a razor knife. The PE dissolved slowly in tetrahydrofuran (THF) but was apparently insoluble in diethyl ether. When heated the PE softened as heated (up to 80°C) but retained its rubbery nature.

The AC conductivities of this material, at the ambient and elevated temperatures, are shown in Table I. The conductivities increase with increasing temperature due, at least in part, to the decreasing viscosity of the polymer. Similar measurements were made using PE formed in a like manner from 2.0M LiAsF₆ in dioxolane. The PE appeared softer and the conductivity values were generally greater. An additional reaction using 1.0M LiAsF₆ electrolyte gave even a softer PE and higher conductivities. The increase in conductivity with decrease in salt concentration may result from the lowered viscosity, i.e., molecular weight, of the polymer formed. This would imply that LiAsF₆ promotes polymerization and that the degree of polymerization is dependent on LiAsF₆ concentration. On the other hand, dioxolane polymerizes readily as a result of chemical initiation in the absence of any salt (6). Thus the differences in viscosity may be due to other factors, such as the degree of salt complexation. Polymerization may also be initiated by oxygen and heat. When this occurs, the precursor

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electrolyte containing the most LiAsF₆ again gives the most rigid and rubber-like polymer.

Table 1. AC and DC* Conductivities (S/cm) of LiAsF₆/Dioxolane Solutions Polymerized using DDQ

Temp.	2.5m LiAsF ₆	2.0m LiAsF ₆	1.0m LiAsF ₆
rt	2 x 10 ⁻⁶ (1 x 10 ⁻⁶)	10 x 10 ⁻⁶ (3 x 10 ⁻⁶)	40 x 10 ⁻⁶ (30 x 10 ⁻⁶)
40°C	10 x 10 ⁻⁶	30 x 10 ⁻⁶	-
50°C	30 x 10 ⁻⁶	-	-
60°C	100 x 10 ⁻⁶	100 x 10 ⁻⁶	-

*DC in parentheses

EC Preparation of The PE.—The EC oxidation of dioxolane can be seen in Figure 1 which shows the cyclic voltammetric evaluation of LiAsF₆/dioxolane electrolyte at its oxidative limit. Using a freshly polished electrode, the first sweep shows appreciable currents due to the oxidation of dioxolane. However, on subsequent sweeps the oxidative currents are greatly diminished. The lessened currents are, of course, due to the polymer film protecting the bulk electrolyte from further oxidation.

In the initial preparations of the PE, the PE was formed in a cell (Cell I) containing liquid electrolyte in excess of the amount that would be incorporated into the PE film. Polymerizations were typically initiated potentiostatically, with the polymerization being terminated by the removal of the anode from the cell. In a typical preparation, polymerization was initiated in a 2.5m precursor solution by potentiostating the anode at 3.5V (vs. Li) for 15 s. During this time, the current jumped to ~30 mA and decreased to 2 mA. The anode was immediately removed from the cell and was covered with a 0.1 cm PE film. The film, when removed from the substrate, is transparent and flexible. Similar preparations were done using 2.0m precursor electrolyte using 3.0 and 3.5V initiation potentials (2.5V gave no polymerization). At rt, these films had AC conductivities in the range 6-8 x 10⁻⁶ S/cm with the DC conductivities somewhat decreased (3 x 10⁻⁶ S/cm).

It seems likely that the presence of residual volatile species would have a significant positive effect on conductivity. Therefore, films similar to those above were prepared and held under vacuum for 1 hour, the conductivities observed were the same as those quoted above.

The conductivities of the PE films formed using EC initiation are comparable in magnitude to those prepared using DDQ initiation, see Table 1, with the AC conductivities generally larger than the DC conductivities. The smaller values of the DC conductivities reflects the contribution of Li ion transport to the total conductivity and the resistance at the Li/PE interface. The conductivities observed are somewhat greater than those observed for PEO and PPO polymer electrolytes (1).

Improved PE films could be made using a thin cell in which the PE is allowed to completely fill the cell (Cell II). Moreover, this thin cell could then be used as a conductivity cell as well. Polymerizations were initiated galvanostatically and the progress of the polymerization followed by monitoring the resistance (from complex impedance measurements) across the cell. The results using a 0.025 cm cell are shown in Figure 2. The conductivity of the electrolyte drops rapidly immediately after initiation and approaches a plateau at 24 h. The resulting film is transparent and elastic; and has an AC conductivity of 3 x 10⁻⁶ S/cm.

Conductivity values were measured as a function of temperature up to 100°C. The conductivity at this temperature was 7 x 10⁻⁴ S/cm with good short term thermal stability indicated by the reproducibility of conductivities remeasured at lower temperatures. As the PE is heated it appears to soften at 80°C with melting occurring in the range 120-130°C. At this temperature discoloration occurs, presumably due to the thermal instability of LiAsF₆.

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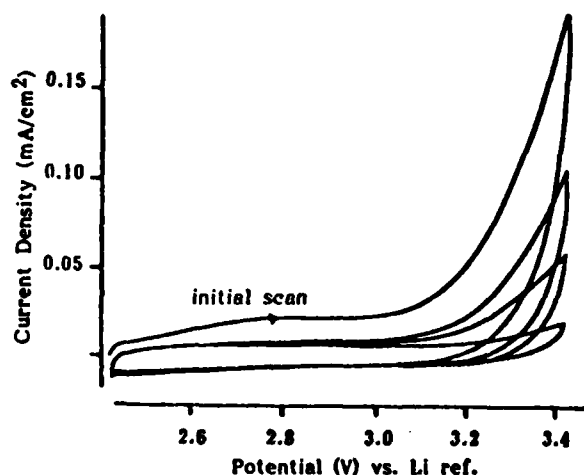


Fig. 1. Repeated CV scans using a Pt electrode in 2.5m LiAsF₆ dioxolane at 100 mV/s.

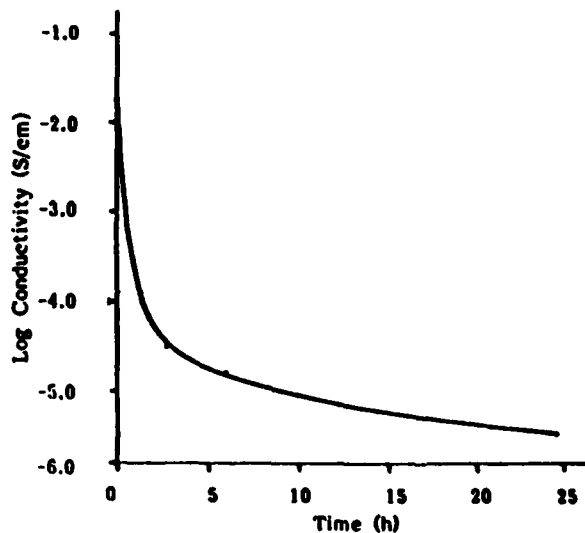


Fig. 2. Log conductivity vs. elapsed time after initiation of polymerization of 2.5m LiAsF₆ in dioxolane. (The initial point was determined in a separate cell.)

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